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(30) Priority Data:

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#### Published

With international search report.

Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.

(54) Title: PERFUME COMPOSITIONS

(57) Abstract

A perfume composition contains at least 40 % by weight of at least three out of six defined perfume ingredient categories of hydroxylic materials, ketones, aldehydes, ethers, esters and nitriles. The perfume composition exhibits a deodorant effect, and is suitable for use in deodorant products, personal products and laundry treatment products.

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#### **PERFUME COMPOSITIONS**

This invention relates to perfume compositions, that is to say compositions of fragrance materials, to personal products and other products containing such perfume compositions, and to the use of such perfume compositions to give a deodorant effect.

5 EP-B-3172, EP-A-5618, US-A-4304679, US-A-4322308, US-A-4278658, US-A-4134838. US-A-4288341, USA-4289641 and US 4 906 454 all describe perfume compositions which exhibit a deodorant action, (i.e. addresses the problem of human body odour, particularly that of the axillae) either when applied to human skin using a cosmetically acceptable vehicle or when included in a detergent product or fabric conditioning product used in laundering of textiles. EP-B-147191 and US-A-4663068 describes deodorant perfume compositions which are stable in the presence of bleaching materials.

A difficulty with the perfume compositions disclosed in these documents is that they generally include appreciable quantities of relatively high molecular weight perfume components which help to extend the effective lifetime of deodorant action following product application, but 15 which tend to have less perfume impact and to exhibit odour characteristics which span a finite range. This limitation on perfume composition represents a compromise between long term deodorant efficacy and optimal hedonic performance.

We have now found that deodorant perfumes can be made by the use of materials from certain specified categories of perfume materials which makes it possible to obtain fragrances 20 containing lower quantities of high boiling components while also obtaining good long term deodorant properties. Forms of this invention can deliver a deodorant performance which improves on that obtained from compositions exemplified in the prior documents above.

Accordingly, the present invention provides a perfume composition comprising at least 40% in total by weight of at least three of the following six categories of perfumery ingredients;

25 a) at least 1.0%, preferably at least 5% and generally not more than 50% by weight of the perfume composition comprises one or more hydroxylic materials of general formula

#### R¹OH

- having an octanol-water partition coefficient within the range of 2.5 to 3.6 (in logarithmic form), and wherein the group R¹ is a hydrocarbyl radical containing no more than one olefinic double bond, and comprising aromatic or aliphatic groups, and mixtures thereof, and which may be cyclic or acyclic, straight chained or branched, and optionally substituted with other groups,
- at least 1.0%, preferably at least 5% and generally not more than 30% by weight of
   the perfume composition of one or more ketones selected from ketones of general formula

#### R'CO R2

having an octanol-water partition coefficient within the range of 3.0 to 4.1 (in logarithmic form), and wherein the groups R¹ and R² are independently hydrocarbyl radicals which may comprise aromatic or aliphatic groups, and mixtures thereof, and may be cyclic or acyclic, straight chained or branched, and optionally substituted with other groups,

c) aldehydes of general formula

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#### R'CHO

having an octanol-water partition coefficient within the range of 2.0 to 4.4 (in logarithmic form), and wherein the group R¹ is a hydrocarbyl radical which may comprise aromatic or aliphatic groups, and mixtures thereof, and which may be cyclic or acyclic, straight chained or branched, and optionally substituted with other groups, with the proviso that if no aromatic group is present then R¹ comprises an olefinic double bond,

15 d) ethers of general formula

#### R¹O R²

having an octanol-water partition coefficient within the range 3.0 to 4.0 (in logarithmic form), and wherein the groups  $R^1$  and  $R^2$  are independently hydrocarbyl radicals which may comprise aliphatic or aromatic groups, and mixtures thereof, and which may be straight chained or branched and optionally substituted with other groups, with the proviso that at least one of  $R^1$  and  $R^2$  comprises an olefinic double bond,

e) esters of general formula

#### R¹CO, R²

having an octanol-water partition coefficient within the range 2.6 to 4.3 (in logarithmic form), and wherein the groups R¹ and R² are independently hydrocarbyl radicals which may comprise saturated aliphatic or aromatic groups, and mixtures thereof, and which may be straight chained or branched, cyclic or acyclic, and optionally substituted with other groups, and

f) nitriles of general formula

30 R'CN

having an octanol-water partition coefficient within the range 3.0 to 4.4 (in logarithmic form), and wherein the group R¹ is a hydrocarbyl radical comprising an olefinic double bond, which may comprise aliphatic or aromatic groups, and mixtures thereof, and which may be straight chained or branched, cyclic or acyclic and optionally substituted with other groups.

The invention also provides a deodorant product comprising a perfume composition as defined above.

The invention further provides the use, as a deodorant, of a perfume composition and a deodorant product as defined above.

The term 'perfume material (or ingredient)' is herein taken to represent materials which may be acceptably employed within fragrances to provide an odour contribution to the overall hedonic performance of the fragrance. Typically, such materials will be generally recognised as possessing odours in their own right, and will be relatively volatile, and characterised by 5 molecular weights within the range of around 100 to 300 amu.

The concentration of perfume materials or ingredients referred to herein is relative to the total concentration of perfume components present in the composition, ie excludes, for example, the presence of any optional diluent.

The octanol-water partition coefficient (or its common logarithm to base 10, 'logP') is well 10 known in the literature as an indicator of hydrophobicity and water solubility (see Hansch and Leo, *Chemical Reviews*, 526 to 616, (1971), 71; Hansch, Quinlan and Lawrence, *J.Organic Chemistry*, 347 to 350 (1968), 33). Where such values are not available in the literature they may be measured directly, or approximately estimated using mathematical algorithms. Software providing such estimations are available commercially, for example 'LogP' from 15 Advanced Chemistry Design Inc.(ACD). For the purposes of the present invention the results obtained using ACD software are preferred.'

A perfume composition according to the present invention has the following preferred features, either singly or in any combination;

(i) hydroxylic materials of category (a) which are one or more of

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Citronellol

Dimethylheptanol

4-(4'-hydroxy-4'-methylpentyl)cyclohex-3-enecarbaldehyde 3-(4'-hydroxy-4'-methylpentyl)cyclohex-3-enecarbaldehyde Tetrahydrolinalol,

- 25 (ii) aldehydes of category (c) which comprise less than two hydrogen atoms in the position directly adjacent to the formyl functional group,
  - (iii) ethers of category (d) in which R¹ comprises an alicyclic or aromatic ring, and
  - (iv) esters of category (e) in which R¹ or R² comprises an alicyclic or aromatic ring.

In certain instances, it may be that materials are capable of classification into more than one 30 category, for example, 4-(4'-hydroxy-4'-methylpentyl)cyclohex-3-enecarbaldehyde comprises both hydroxyl and formyl functional groups and hence could be a member of category (c) as well as (a). In such cases the material is deemed to be within the first named category, that is, in (a) before (b), in (d) before (e) and so forth. Acetals are considered herein as ethers. With regard to the essential oils, synthetic oils and complex mixtures common within the perfumery 35 business, the above rules must be applied to their individual constituents.

Preferred perfumes comprise at least 50%, more preferably at least 60%, and particularly at least 75% by weight of perfume ingredients as described herein. The perfume composition preferably comprises at least 4, more preferably at least 5, and particularly all 6 classes of perfumery ingredients as described herein.

5 The invention is directed to perfume compositions and to consumer products which provide a deodorant action when applied to the body within a cosmetically acceptable vehicle. Suitable deodorant products include, but are not limited to, deodorants and antiperspirants including different physical forms such as roll ons, gels, sticks, and aerosols, other personal products such as deocolognes, talcum powders, hand creams, lotions, skin and hair conditioners, 10 sunscreens, soaps, shampoos, and shower gels.

The perfumes described herein may also be usefully employed for deodorant properties in other product areas, for example in detergent and household products such as laundry powders, laundry liquids, rinse conditioners, and household cleaning compositions. Perfumes of the inventions may also be incorporated into textiles directly during manufacture using 15 techniques known in the art, to provide long lasting deodorant protection. It is also known in the art to carry or encapsulate perfumes within other materials such as porous solids or polymeric matrices, in order to provide extended lifetimes, and to provide the possibility of triggered release, for example, during perspiration. Such techniques are applicable within the scope of the present invention.

20 The invention is illustrate by the following examples.

#### **EXAMPLES**

Table 1 presents a representative list of perfume materials falling within the above categories, together with comparative examples of commonly used perfume ingredients which do not fall within the categories.

25 Table 2 presents a perfume composition 'A' falling within the scope of the invention, together with a comparative example 'B' comprising deodorant perfume constructed from the knowledge in the prior documents above, and comprising significant quantities of high boiling low impact perfume materials.

Tables 3 to 5 illustrate the use of perfumes of the invention within deodorant products. Their 30 preparation is well known to those skilled in the art.

Perfumes A and B were tested for long term deodorant action in the underarm roll-on product whose composition is given in Table 5, using an Odour Reduction Value test as generally described in US-A-4278658. The results of this 24 hour deodorant test are given in Table 6,

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and demonstrate that perfume 'A' provides at least as effective long term deodorancy as the perfume B built on musks and other substantive ingredients.

<u>Table 1</u>
Examples of Ingredient Categorisation

Material	Category
1,3,4,6,7,8-hexahydro-4,6,6,7,8,8-hexamethylcyclopenta[g] -2-benzopyran	ex
1-(3,5,5,6,8,8-hexamethyl-5,6,7,8-tetrahydro-2-naphthalenyl) -1-ethanone	ex
1-Methyl-2-propenyl-4-isopropyl benzene	ex
2 - tert - Butylcyclohexyl acetate	е
2,4 - dimethylcyclohex - 3 - ene - 1 - carbaldehyde	С
2-Ethyl-4-(2,2,3-trimethyl-3-cyclopenten-1-yl)-2-buten-1-ol	ex
2-methyl-3-(4'-(1''-methylethyl)phenyl)propanal	C .
4-(4'-hydroxy-4'-methylpentyl)cyclohex-3-enecarbaldehyde	а
8,8-Dimethyl-7-(1-methylethyl)-6,10-dioxaspiro[4.5]decane	d
Acetyl di-isoamylene	b
Benzyl salicylate	ex
Cedrenyl acetate	ex
Citronellol	а
Citronellyl nitrile	f
Coumarin	ex
Diethyl phthalate	ex
Diethyldimethylcyclohex-2-en-1-one	b.
Dihydrojasmone	b
Dihydrojasmone	b
Dimethyl benzyl carbinyl acetate	е
Dimethyl heptan-1-ol	а
Dipropylene glycol	ex
Florocylene	ex
Heliotropin	ex
Hexyl cinnamic aldehyde	ex

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Hexyl salicylate	ex
lonones	b
Methyl ionones	ex
Methyl isoeugenol	d
Phenylethyl alcohol	ex
Tetrahydrolinalol	а
Undecalactone, gamma-	· e
Vanillin	ex

Note: 'ex' = excluded from classification

<u>Table 2</u>
Compositions of perfumes A and B

Mala		w/w%	Category
Material	Α	B	
1,3,4,6,7,8-hexahydro-4,6,6,7,8,8-hexamethylcyclopenta[g] -2-benzopyran		6.00	
2 - tert- butylcyclohexyl acetate	2 .00		е
2,4 - dimethylcyclohex -3-ene- 1- carbaldehyde	0.2		С
2,6,10-trimethylundec-9-enal	0.5		
2-methyl-3-(4'-(1"-methylethyl)phenyl)propanal	3.00		С
3a,4,5,6,7,7a-hexahydro-4,7-methano-1H-inden-6-yl- propanoate		3.00	
8,8-Dimethyl-7-(1-methylethyl)-6,10-dioxaspiro[4.5]decane Acetyl di-isoamylene	10.00 6.00		d b
Benzyl acetate		6.00	
Benzyl salicylate		6.00	
Cassis base AB2967 (Q)	0.80		
Cedrenyl acetate		8.00	
Cervolide	4.00		
Decanal		0.2	
Diethyl phthalate	7.00	6.00	
Diethyldimethylcyclohex-2-en-1-one	3.00		b
Dihydrojasmone	0.500		b
Dimethyl benzyl carbinyl acetate	2.00		е
Dipropylene glycol	9.00	1.8	
Geranium ABQ5629 (Q)		4.00	
Hexyl cinnamic aldehyde	6.00	6.00	
lonone, alpha-	10.00		b
Lavandin oil		14.0	
Lixetone (Q)		8.00	
Methyl dihydrojasmonate	15.5		

Methyl ionone, alpha iso-		8.00	
Methyl isoeugenol	2.00		d
Muguet base AB1951 (Q)	6.00	5.00	
Rosemary oil (Tunisian)		3.00	
Terpinyl acetate		8.00	
Tetyrahydrolinalol	12.00		a
Undecalactone, gamma	0.5		e
Extralide(Q)		3.00	

Note: materials marked 'Q' are available from Quest International.

<u>Table 3</u> Deodorant stick

Ingredient	Weight %
Ethanol	44.0
Sodium Stearate	7.0
Propylene glycol	11.0
Perfume	1.0
PEG-6-Caprylic/capric	12.0
glycerides	
Glycerin	5.0
Water	20.0

Table 4

#### 5 Deodorant aerosol

Ingredient	Weight%
Isopropyl myristate	3.0
Propellants	to 100%
Fumed silica	0.25
Perfume	1.5

1.00

to 100%

Perfume

Water

	Weight%	Weight%
Ingredient		
Ethanol		60.0
Klucel MF		0.65
Cremophor RM410	-	0.5
Bentone gel IPM (Rheox Inc.)	27.0	
Siliocone fluid DC344 (Dow Corning)	to 100%	
Aluminium chlorhydratė powder	20.0	

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<u>Table 6</u>
Results of 24 hr underarm deodorant efficacy test

Average panel score perfume A	1.85
Average panel score perfume B	1.89
Control panel score	2.41
Odour Reduction Value perfume A	0.56
Odour Reduction Value perfume B	0.52
Odour Reduction Value as percentage of control score - perfume A	23.2%
Odour Reduction Value as percentage of control score - perfume B	21.6%

0.75

Difference for significance @95% 0.18

<sup>5</sup> The results show that perfume A yields excellent deodorancy effect, whilst having improved hedonic properties.

#### **CLAIMS**

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1. A perfume composition comprising at least 40% in total by weight of at least three of the following six categories of perfumery ingredients;

 a) at least 1.0%, preferably at least 5% and generally not more than 50% by weight of the perfume composition comprises one or more hydroxylic materials of general formula

#### R'OH

having an octanol-water partition coefficient within the range of 2.5 to 3.6 (in logarithmic form), and wherein the group R¹ is a hydrocarbyl radical containing no more than one olefinic double bond, and comprising aromatic or aliphatic groups, and mixtures thereof, and which may be cyclic or acyclic, straight chained or branched, and optionally substituted with other groups,

 at least 1.0%, preferably at least 5% and generally not more than 30% by weight of the perfume composition of one or more ketones selected from ketones of general formula

#### R'COR2

having an octanol-water partition coefficient within the range of 3.0 to 4.1 (in logarithmic form), and wherein the groups R¹ and R² are independently hydrocarbyl radicals which may comprise aromatic or aliphatic groups, and mixtures thereof, and may be cyclic or acyclic, straight chained or branched, and optionally substituted with other groups,

c) aldehydes of general formula

#### R'CHO

having an octanol-water partition coefficient within the range of 2.0 to 4.4 (in logarithmic form), and wherein the group R¹ is a hydrocarbyl radical which may comprise aromatic or aliphatic groups, and mixtures thereof, and which may be cyclic or acyclic, straight chained or branched, and optionally substituted with other groups, with the proviso that if no aromatic group is present then R¹ comprises an olefinic double bond,

30 d) ethers of general formula

#### R¹O R²

having an octanol-water partition coefficient within the range 3.0 to 4.0 (in logarithmic form), and wherein the groups  $R^1$  and  $R^2$  are independently hydrocarbyl radicals which may comprise aliphatic or aromatic groups, and mixtures thereof, and which may be straight chained or branched and optionally substituted with other groups, with the proviso that at least one of  $R^1$  and  $R^2$  comprises an olefinic double bond,

e) esters of general formula

#### R'CO, R2

having an octanol-water partition coefficient within the range 2.6 to 4.3 (in logarithmic form), and wherein the groups R¹ and R² are independently hydrocarbyl radicals

which may comprise saturated aliphatic or aromatic groups, and mixtures thereof, and which may be straight chained or branched, cyclic or acyclic, and optionally substituted with other groups, and

f) nitriles of general formula

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#### R¹CN

having an octanol-water partition coefficient within the range 3.0 to 4.4 (in logarithmic form), and wherein the group R¹ is a hydrocarbyl radical comprising an olefinic double bond, which may comprise aliphatic or aromatic groups, and mixtures thereof, and ¬ which may be straight chained or branched, cyclic or acyclic and optionally substituted with other groups.

2. A perfume composition according to claim 1 wherein the hydroxylic materials of category (a) comprise one or more of the following materials

Citronellol

Dimethylheptanol

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4-(4'-hydroxy-4'-methylpentyl)cyclohex-3-enecarbaldehyde 3-(4'-hydroxy-4'-methylpentyl)cyclohex-3-enecarbaldehyde Tetrahydrolinalol.

- 3. A perfume composition according to either one of claims 1 and 2 wherein the aldehydes of category (c) comprise less than two hydrogen atoms attached to the carbon 20 atom adjacent to the carbonyl function.
  - 4. A perfume composition according to any one of the preceding claims wherein the ethers of category (d) comprise an alicyclic or aromatic ring within R¹.
  - 5. A perfume composition according to any one of the preceding claims wherein the esters of category (e) comprise an alicyclic or aromatic ring within R¹ or R².
- 25 6. A perfume composition according to any one of the preceding claims comprising at least 50% by weight of specified ingredients.
  - 7. A perfume composition according to any one of the preceding claims comprising at least 60% by weight of specified ingredients.
- 8. A perfume composition according to any one of the preceding claims comprising at 30 least 75% by weight of specified ingredients.
  - 9. A deodorant product comprising a perfume composition defined in any one of the preceding claims.
  - 10. A deodorant product according to claim 9 wherein the product is a personal product including shampoos, creams, lotions, conditioners, soaps and talcs.
- 35 11. A deodorant product according to claim 9 wherein the product is a laundry treatment product including detergents and rinse conditioners.
  - 12. The use, as a deodorant, of a perfume composition defined in any one of claims 1 to 8, and of a deodorant product defined in any one of claims 9 to 11.

# INTERNATIONAL SEARCH REPORT

Inter anal Application No PCT/GB 99/02164

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A. CLASS IPC 7	SIFICATION OF SUBJECT MATTER A61K7/46 A61K7/32		
According t	to International Patent Classification (IPC) or to both national classif	ication and IPC	
	SEARCHED	· · · · · · · · · · · · · · · · · · ·	
Minimum de IPC 7	locumentation searched (classification system followed by classifica A61K	ation symbols)	
Documenta	ation searched other than minimum documentation to the extent that	such documents are included in the fi	elds searched -
Electronic	data base consulted during the international search (name of data t	pase and, where practical, search term	s used)
C. DOCUM	ENTS CONSIDERED TO BE RELEVANT		
Category °	Citation of document, with indication, where appropriate, of the r	elevant passages	Relevant to claim No.
X	WO 94 24999 A (QUEST INTERNATION AL.) 10 November 1994 (1994-11-1 examples 1-3		1-5,9-12
X	GB 2 013 493 A (UNILEVER) 15 August 1979 (1979-08-15) page 8, line 92 - line 114 page 9, line 90 - line 108 & US 4 278 658 A cited in the application		9-11
Α	EP 0 005 618 A (UNILEVER PLC ET 28 November 1979 (1979-11-28) cited in the application page 6, line 29 -page 7, line 50 examples 1-5	·	
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X Funi	her documents are listed in the continuation of box C.	X Patent family members are	listed in annex.
"A" document defining the general state of the art which is not considered to be of particular relevance "F" earlier document but published on or after the international		"T" later document published after the or priority date and not in conflicted to understand the principle invention  "X" document of particular relevance.	ct with the application but e or theory underlying the e; the claimed invention
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	11 October 1999	03/11/1999	
Name and r	mailing address of the ISA  European Patent Office, P.B. 5818 Patentlaan 2  NL - 2280 HV Rijswijk  Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,  Fax: (+31-70) 340-3016	Authorized officer  Alvarez Alvare	ez, C





inter nal Application No PCT/GB 99/02164

0.70=====	Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
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A	DATABASE WPI Week 199829 Derwent Publications Ltd., London, GB; AN 1998-328365 XP002119810 & JP 10 120541 A (BEIERSDORF AG ET AL), 12 May 1998 (1998-05-12) abstract	1-12	
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# INTERNATIONAL SEARCH REPORT

lı .national application No.

PCT/GB 99/02164

Box I Observations where certain claims were found unsearchable (Continuation of item 1 of	of first sheet)
This International Search Report has not been established in respect of certain claims under Article 17(2)(a) for the	ne following reasons:
Claims Nos.:     because they relate to subject matter not required to be searched by this Authority, namely:	· .
2. X Claims Nos.: because they relate to parts of the International Application that do not comply with the prescribed required an extent that no meaningful International Search can be carried out, specifically: see FURTHER INFORMATION sheet PCT/ISA/210	rements to such
Claims Nos.:     because they are dependent claims and are not drafted in accordance with the second and third sentence.	
Box II Observations where unity of invention is lacking (Continuation of item 2 of first sheet)	
This International Searching Authority found multiple inventions in this international application, as follows:	
As all required additional search fees were timely paid by the applicant, this International Search Report searchable claims.	covers all
2. As all searchable claims could be searched without effort justifying an additional fee, this Authority did not of any additional fee.	ot invite payment
3. As only some of the required additional search fees were timely paid by the applicant, this International Scovers only those claims for which fees were paid, specifically claims Nos.:	- Gearch Report
4. No required additional search fees were timely paid by the applicant. Consequently, this International Se restricted to the invention first mentioned in the claims; it is covered by claims Nos.:	arch Report is
Remark on Protest  The additional search fees were accompanied by the No protest accompanied the payment of additional search.	

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#### FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

Continuation of Box I.2

INCOMPLETE SEARCH FOR CLAIMS 1 (TOTALLY) AND 3-12 (PARTIALLY)

Present claims 1 and 3-12 (partially) relate to an extremely large number of possible compounds. Support within the meaning of Article 6 PCT and disclosure within the meaning of Article 5 PCT is to be found, however, for only a very small proportion of the compounds claimed.

In the present case, the claims 1 (totally) and 3-12 (partially) so lack support and the application lacks disclosure, that a meaningful search over the whole of the claimed scope is impossible. Consequently, the search has been carried out for the parts of the claims which appear to be supported and disclosed, namely relating to compounds listed in table 1 (not belonging to category "ex") and table 2 composition A, as components of perfume compositions with deodorant properties, in the amounts and proportions claimed in claim 1.

The applicant's attention is drawn to the fact that claims, or parts of claims, relating to inventions in respect of which no international search report has been established need not be the subject of an international preliminary examination (Rule 66.1(e) PCT). The applicant is advised that the EPO policy when acting as an International Preliminary Examining Authority is normally not to carry out a preliminary examination on matter which has not been searched. This is the case irrespective of whether or not the claims are amended following receipt of the search report or during any Chapter II procedure.

...formation on patent family members

Inter nal Application No PCT/GB 99/02164

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